DEPARTMENT OF MINES.

South Australia.

-RESEARCH AND DEVELOPMENT BRANCH-

R.D. 70.

FLOTATION OF OXIDE MINERALS.

PART 1.

Effect of the Degree of Unsaturation of Linseed Fatty Acid Reagents on flotation efficiency.

by

E.E. Moskovits.
G.D. Sheridan.

ISSUED BY:
T.W. Dalwood.
Chief Superintendent.

Copy No. 3 of 25 copies.

DATE: August, 1957.

This document consists of 14 pages.

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PART 1.

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Effect of the degree of unsaturation of Linseed Fatty Acid reagents on flotation efficiency.

-Abstract-

Tests are reported which show that the flotation efficiency of linseed fatty acid type reagents used at Radium Hill increases as the degree of unsaturation of the fatty acid increases.

Limits are set on the iodine and acid values as a guide to the purchase of reagents but laboratory flotation tests are recommended as a final check.

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1. SUMMARY.

An investigation was carried out to correlate flotation efficiency with degree of unsaturation of fatty acid type reagents.

Fatty acids from linseed oil were used in the test work. The unsaturated acids present in this type of reagents are mainly oleic, linoleic and linolenic acids, the last being the most unsaturated.

Tests were carried out using pure oleic, linoleic and linolenic acids and it was found that flotation efficiency improved with the use of the more unsaturated acid. Three commercial products, having varying contents of these acids, supplied by Meggitts Ltd. showed a trend similar to that indicated by the pure acids.

Many other linseed products were tested with the object of showing that the iodine and acid values can be used as a guide to the suitability of the product as a flotation reagent .

It is considered that these values can be used as a guide only, and the effectiveness of the reagent would have to be confirmed by laboratory flotation tests.

2. INTRODUCTION.

In the course of investigations into the use of fatty acids as flotation reagents, it became apparent that a correlation existed between total unsaturated acid content and metallurgical efficiency. In general, fatty acids having a high degree of unsaturation gave good recoveries. A search was therefore made for suitable products on the local market and, as linseed fatty acids were readily available, these were chosen as the basis of the test work.

The fatty acid product from linseed is subject to considerable variation in composition with consequent variable behaviour in flotation performance.

It seemed desirable to determine which components of the mixed commercial products were the essential flotation reagents and, if possible, to define certain characteristics which would form a basis for the selection of material having adequate flotation properties.

Unfortunately the analytical methods in general use for determining fatty acid composition are subject to considerable uncertainty , even when carried out under carefully controlled conditions. For this reason the composition indicated by the manufacturer may not be a reliable guide to the behaviour of the material as a flotation reagent.

The degree of unsaturation of a fat can be expressed in terms of the iodine value and it was thought that this figure might form a useful criterion in assessing the flotation properties of commercial fatty acid products. Accordingly iodine and acid values were determined for a number of fatty acids, which were also classified according to their flotation efficiencies.

The fatty acid reagents require the addition of other products to ensure complete dispersion and optimum results. For the tests outlined in this report these reagents consisted of peltogen, fuel oil and cresylic acid.

The role of each reagent is complex but an explanation has been suggested and is given in some detail in report MT77 "Flotation of Davidite from Radium Hill ore". (1)

3. MATERIAL EXAMINED.

Various types of linseed fatty acids and linseed oil products as well as the pure major constituents of these linseed products were used in this investigation.

The six basic reagents were as follows:

Oleic Acid.	(commercial grade).
Linoleic Acid.	(experimental).
Linolenic Acid.	(experimental).
Linseed Fatty Acid	S100 Minimum (commercial).
" " "	S100 Average (")
" " "	S100 Maximum (").

The last three products were supplied by Meggitts Limited and were stated to have the following composition.

	S100 Minimum.	S100 Average.	S100 Maximum.
Oleic Acid.	10 percent.	10.5 percent.	12 percent.
Linoleic Acid.	10 "	10.5 "	12 "
Linolenic Acid.	38.5 "	41.0 "	49.5 "

4. EQUIPMENT and ANCILLARY MATERIALS.

The equipment and ore types used were:

Fagergren flotation cell (500 grams capacity).

Standard Radium Hill minus 10 mesh ore, and heavy media concentrate.

5. EXPERIMENTAL PROCEDURE.

All flotation tests were done according to a standard procedure. The ore was ground with reagents to the desired degree of fineness, transferred to the flotation cell and floated for a fixed time.

The pure major components, oleic, linoleic and linolenic acids were first tested. Of these, oleic acid has the lowest and linolenic acid has the highest degree of unsaturation.

Further tests were then carried out using the three linseed products supplied by Meggitts Ltd. These products designated "S100 - Minimum", "S100 - Average" and "S100 - Maximum" contained low, medium and high amounts respectively of unsaturated acids.

Another series of tests was done using samples of various linseed products made available by other manufacturers.

The acid and iodine values of all products tested were obtained with the object of correlating flotation efficiency with the known degree of unsaturation of each product.

In all flotation tests the various linseed fatty type reagents were used in conjunction with three other reagents peltogen, fuel oil and cresylic acid.

The amounts of reagents used for each test are given below in the various sections.

6. CONDITIONS and RESULTS.

6.1 Series 1 - Flotation using pure acids.

This series was carried out to show the effect on flotation efficiency of oleic, linoleic, and linolenic acids when used individually with the three standard reagents. The reagent combinations used for this series are shown in Table 1.

The three tests reported are typical of the many tests carried out with these pure acids.

TABLE 1.

Series 1 - Reagent Combinations.

Test No.	1.	2.	3.
Reagents.	lb/ton.	lb/ton.	lb/ton.
Oleic Acid.	1.0	-	-
Linoleic Acid.	-	1.0	-
Linolenic Acid.	-	-	1.0
Peltogen.	0.75	0.75	0.75
Fuel Oil.	3.0	3.0	3.0
Cresylic Acid.	0.25	0.25	0.25

For these tests the minus 10 mesh fraction of Radium Hill ore was used. The results are given in Table 2.

TABLE 2.

Series 1 - Results.

Test No.	Fraction.	Percent Weight.	U_3O_8 lb/ton.	Percent Distrib. U_3O_8
1.	2nd cleaner concentrate.	6.2	10.8	23.6
	2nd cleaner tailing.	4.8	9.0	15.2
	1st cleaner tailing.	11.0	5.3	20.7
	Rougher tailing.	78.0	1.48	40.5
	FEED	100.0	2.8	100.0
2.	2nd cleaner concentrate.	8.0	11.2	33.0
	2nd cleaner tailing.	3.2	8.3	9.9
	1st cleaner tailing.	11.4	5.8	24.5
	Rougher tailing.	77.4	1.15	32.6
	FEED	100.0	2.7	100.0
3.	2nd cleaner concentrate.	9.7	12.0	40.3
	2nd cleaner tailing.	4.0	9.1	12.7
	1st cleaner tailing.	14.3	4.4	22.0
	Rougher tailing.	72.0	1.01	25.0
	FEED	100.0	2.8	100.0

6.2 Series 2 - Flotation using Meggitts S100 Reagents.

Tests were done using the three S100 type reagents supplied by Meggitts Limited. In these tests two types of ore were used, namely the minus 10 mesh fraction and the heavy media concentrate of the Radium Hill material.

The reagent combinations used for these tests are given in Table 3.

TABLE 3.

Series 2 - Reagent Combinations.
(Reagent quantities expressed as lb/ton of ore).

Test No. Reagents.	4.	5.	6.	7.	8.	9.	10.	11.	12.
S100 Minimum.	1.0	-	-	2.0	-	-	2.0	-	-
S100 Average.	-	1.0	-	-	2.0	-	-	2.0	-
S100 Maximum.	-	-	1.0	-	-	2.0	-	-	2.0
Peltogen.	0.75	0.75	0.75	1.5	1.5	1.5	1.5	1.5	1.5
Fuel Oil.	3.0	3.0	3.0	6.0	6.0	6.0	6.0	6.0	6.0
Cresylic Acid.	0.25	0.25	0.25	0.5	0.5	0.5	0.5	0.5	0.5

Tests 4 to 9 inclusive were done on the minus 10 mesh fraction of Radium Hill ore and the remaining three tests on a mixture of equal proportions of heavy media concentrate and minus 10 mesh ore.

The results of these tests are given in Table 4.

TABLE 4.

Series 2 - Results.

Test No.	Fraction.	Percent Weight.	U_3O_8 lb/ton.	Percent Distrib. U_3O_8.
4.	Cleaner concentrate.	0.6	9.4	1.8
	Cleaner tailing.	12.4	8.4	36.4
	Rougher tailing.	87.0	2.06	61.8
	FEED	100.0	2.9	100.0
5.	Cleaner concentrate.	2.5	11.2	9.8
	Cleaner tailing.	11.6	9.6	39.0
	Rougher tailing.	85.9	1.71	51.2
	FEED.	100.0	2.9	100.0
6.	Cleaner concentrate.	9.1	10.9	37.2
	Cleaner tailing.	12.7	6.4	30.5
	Rougher tailing.	78.2	1.10	32.3
	FEED	100.0	2.7	100.0
7.	2nd cleaner concentrate.	9.9	13.3	41.7
	2nd cleaner tailing.	7.7	11.3	27.6
	1st cleaner tailing.	12.3	4.3	16.7
	Rougher tailing.	70.1	0.63	14.0
	FEED	100.0	3.2	100.0
8.	2nd cleaner concentrate.	9.5	12.9	43.2
	2nd cleaner tailing.	6.8.	10.0	24.0
	1st cleaner tailing.	14.3	3.7	18.7
	Rougher tailing.	69.4	0.57	14.1
	FEED	100.0	2.8	100.0
9.	2nd cleaner concentrate.	14.3	11.9	59.5
	2nd cleaner tailing.	6.8	7.2	17.1
	1st cleaner tailing.	17.3	2.2	13.3
	Rougher tailing.	61.6	0.47	10.1
	FEED.	100.0	2.9	100.0
10.	2nd cleaner concentrate.	11.1	26.0	28.4
	2nd cleaner tailing.	2.6	26.0	6.6
	1st cleaner tailing.	27.5	19.2	51.8
	Rougher tailing.	58.8	2.30	13.2
	FEED	100.0	10.2	100.0

Table 4 (continued).

Test No.	Fraction.	Percent Weight.	U_3O_8 lb/ton.	Percent Distrib. U_3O_8
11.	2nd cleaner concentrate.	21.0	26.4	55.8
	2nd cleaner tailing.	10.7	22.2	24.0
	1st cleaner tailing.	8.6	10.3	8.9
	Rougher tailing.	59.7	1.84	11.3
	FEED	100.0	9.9	100.0
12.	2nd cleaner concentrate.	23.4	26.0	58.7
	2nd cleaner tailing.	6.4	20.5	12.6
	1st cleaner tailing.	16.7	12.1	19.4
	Rougher tailing.	53.5	1.82	9.3
	FEED	100.0	10.4	100.0

6.3 Series 3 - Flotation using Various Linseed Products.

A selection of commercially available linseed products was tested in similar manner to the reagents in Series 1 and 2. For the sake of simplicity the flotation efficiency of each of these reagents, taking into account the concentrate grade and uranium distribution in the concentrate, is expressed as "low", "medium" or "high" (see table 5). No assays have been quoted but the rating is derived from their behaviour in numerous tests.

6.4 Flotation Efficiency and Degree of Unsaturation.

The previous three sections cover tests in which various linseed fractions having a wide range of unsaturation were examined.

There are three main values from which the fatty acid composition can be determined, namely:-

(a) Saponification value.

(b) Acid value.

(c) Iodine value.

The saponification value is obtained by boiling the oil with an excess of potassium hydroxide. It gives an indication of the total fatty material present, including both free and combined fatty acids.

The acid value is obtained by a titration of the oil with potassium hydroxide. The difference between the above two determinations will thus give a measure of the glycerides etc. present.

The acid value is used by the manufacturers as a routine control figure for maintaining uniform composition.

The iodine value indicates the degree of unsaturation of the fatty acid constituents. Pure oleic acid has a theoretical iodine value of 90. Values over this, therefore, indicate the presence of the diene derivative, linoleic acid (IV = 180) and the triene derivative (IV = 270).

From a study of the iodine values and the unsaturated fatty acid content an approximate estimate of the quantities of each unsaturated fatty acid present can be found.

The iodine and acid values were determined for each of the reagents tested. The various reagents were then grouped according to their flotation efficiency into three categories, "low", "medium" and "high", as shown in Table 5 which lists these classifications together with the iodine and acid values.

TABLE 5.

Flotation Efficiency and Degree of Unsaturation of Reagents.

Reagents.	Iodine Value.	Acid Value.	Flotation Efficiency.
Fatty acid residue.	48	178	Low
Raw linseed oil.	83	160	Low
Linseed core oil.	128	177	Medium
Linseed fatty acid (Type L.R.O.)	130	160	Medium
Linseed core oil precipitate.	140	227	Medium
Recovered linseed oil (Type 1).	144	142	Medium
S100 Minimum.	150	161	Medium
S100 Average.	151	153	Medium
Recovered Linseed Oil (Type 2).	154	190	Medium
Linseed fatty acid (Type L.O).	121	170	High
Linseed fatty acid (Type L.C.O).	137	149	High
Linseed fatty acid (Type L.F.A.)	149	186	High
Linseed fatty acid Mixture.	161	186	High
S100 Maximum.	165	183	High
Linseed fatty acid (Ex. Rad. Hill).	166	192	High
Fatty Acid Condensate.	169	203	High.

A study of these results and a knowledge of the behaviour of the reagent enabled upper and lower limits to be placed on the iodine and acid values which would almost certainly ensure that the reagent would be suitable for use as a flotation reagents.

7. DISCUSSION OF RESULTS.

7.1 Flotation using pure acids.

The results of these tests are given in section 5.1

The tests were carried out using "starvation" amounts of reagents since past experience has shown that differences are accentuated by this method.

The theoretical iodine values for the three acids tested are:

Oleic acid.	Iodine Value	90.
Linoleic Acid.	" "	180
Linolenic Acid.	" "	270

and the results in Table show an increase in flotation efficiency with the more unsaturated acids.

7.2 Flotation using Meggitts S100 Reagents.

As shown under "Material Examined" these three reagents varied in the total content of unsaturated fatty acids. The results in Table 4 show the same trend as that indicated by the pure acids.

Tests 4, 5 and 6 were carried out using starvation amounts of reagent and indicate more markedly the difference between the three reagents.

7.3 Flotation Efficiency and Degree of Unsaturation.

Table 5 summarizes in simplified form the correlation between flotation efficiency and degree of unsaturation.

As can be seen from this table a relationship exists between flotation efficiency and iodine value. The results of the tests indicate that a linseed fatty acid product having an iodine value of 160 or over and an acid value of 180 or over should fulfil requirements as a flotation reagents.

However, exceptions are evident which could be due to the presence of components in the reagents which affect iodine values and/or flotation efficiency. This explanation would apply to products of low iodine value giving good flotation results and reagents with high iodine values giving poor results.

It is known that factors such as the presence of resin acids or of conjugated diene compounds require the selection of a special method for the determination of iodine value.

It is considered however, that the results shown in Table 5 show a similar trend to that which is evident in the tests of series 1 and 2.

8. OBSERVATIONS and CONCLUSIONS.

While it is evident that the degree of unsaturation of a reagent enhances its value in the flotation of oxide minerals, the determination of the degree of unsaturation by general analytical methods is subject to inaccuracies. Therefore, although the iodine and acid values of a reagent can be taken as a guide, they should not be used as the only criteria in the assessment of the comparative merits of unsaturated compounds.

It is considered that, for specification purposes at Radium Hill, minimum values of 150 and 170 for iodine and acid value respectively, may be quoted to the suppliers and that the final choice of product should be decided on the results of a laboratory flotation tests.

REFERENCE:

(1) Flotation of Davidite from Radium Hill Uranium Ore. Department of Mines - Metallurgical Report MT77 September, 1954.

APPENDIX:

Analytical Methods.

1. Iodine Value.

The iodine value of a fat is defined as "the number of grams of iodine absorbed by one hundred grams of the fat".

A solution of iodine monochloride (Wij's solution) is used. This solution is very unstable and must be freshly prepared for a group of determinations.

Weigh a small container, add about 0.25 grams of the fat and reweigh.

Place the container and fat in a stoppered bottle - Bottle "A".

Add 5 ml carbon tetrachloride and shake to dissolve fat.

Run in exactly 25 ml of Wij's solution from a burette.

Agitate the contents by a rotary motion to ensure that they are thoroughly mixed, and then allow the bottle to stand for 30 minutes.

Repeat the above procedure with another bottle - Bottle "B", but omit the fat.

To each bottle add 5 ml of a solution of potassium iodine (10 percent). Wash to stopper with distilled water and allow washings to run into the bottle.

Add 50 ml of distilled water.

Titrate with N/10 sodium thiosulphate, using freshly prepared starch solution for an indicator.

2. Calculation.

Weight of container plus fat	: 1.75 grams.
" " "	: 1.50 grams.
Weight of fat.	: 0.25 grams.

Bottle "A" required 10.2 ml of N/10 $Na_2S_2O_3$ solution.

Bottle "B" " 27.7 ml " " "

∴ The halogen absorbed by 0.25 gram of fat is equivalent to 17.5 ml of N/10 $Na_2S_2O_3$ that is,

$\frac{17.5 \times 12.7}{100}$ grams of iodine.

∴ 100 grams of fat would absorb $\frac{17.5 \times 12.7 \times 100}{1000 \times 0.25}$.

:88.9 grams iodine.

The iodine value is 88.9